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INVESTIGATION AT VARIOUS TEMPERATURES OF THE SURFACE TENSION OF AQUEOUS SOLUTIONS OF LOWER ALCOHOLS

> B. Ya. Teytelbaum, T. A. Gortalova, Ye. Ye. Sidorova, Kazan' Affiliate, Acad Sci USSR, Chem Inst imeni A. Ye. Arbuz.v

Comment: This is fundamental research which has a bearing on the action of surface active compounds, particularly the influence of temperature changes on their activity. Relationships discovered in work of this type are applicable in connection with the dispersion of liquids in the form of aerosols, foams, emulsions, etc. They are pertinent to studies of wetting capacity and detergency. It may or may not be an accident that the work in question is being carried out at an institute that does a considerable amount of research on toxic agents (organophosphorus derivatives, i.e., a class of compounds which includes insecticides and nerve poisons). Such toxic agents must be effectively dispersed to exert their mex. imum effect. Work on surface activity and surface active agents is conducted at practically every major chemical research institute; the uses to which the results are put depend entirely on the intentions of the people who use them. However, there is a potential and possibly actual connection between the two lines of re-

Aqueous solutions of alcohols have been repeatedly subjected to investigations of the surface tension 6 (1-9). Nevertheless, we have not found any published data on measurements of surface tensions at various temperatures. This made it impossible for us to calculate the values of temperature coefficients

X = 27 or, in other words, of unit entropies of the surface of the solutions. Knowledge of these values, which represent, as we have shown (10), one of the fundemental thermodynamic characteristics of the surface film, is essential for estimating the type of interrelationship that exists between the components present in

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Investigation of surface tensions of aqueous alcohol solutions at various temperatures is also of interest from the standpoint of the foam-forming capacity of the solutions in question. We found earlier that one may determine from temperature-surface tension curves of systems consisting of two organic liquids in our opinion, is a factor which brings about formation of foam.(11)

The surface tension measurements were carried out by determining within intervals of 50 the maximum pressure at which bubbles were formed. The temperature was held constant with a precision of 0.1°. The alcohols used were dehydrated by boiling them with magnesium filings. The mixtures were prepared by the volume method. Their composition is indicated in volume 3. It is well known that among the alcohols studied, n-butyl alcohol and isobutyl alcohol, which are sparingly soluble in water, exhibit a pronounced tendency to produce foams when present in the form of aqueous solutions.

The most general case of limited solubility of liquids is characterized, as is well known, by the existence of two critical solubility points. For instance, these points appear in the system water-nicotine. In any such system, the increased solubility brought about by a lowering of the temperature is due to a reduction of the thermal dissociation of complexes (e.g. hydrates) which the components form with each other (13). These complexes act as homogenizers in systems where there is separation due to great differences between the surface tensions of the components(14).

In some systems, a closed solubility curve cannot be completely achieved by reason of ensuing crystallization or because the critical point of evaporation has been reached. However, in a number of mixtures forming systems of this kind, separation disappears when the temperature is raised or lowered. Aqueous solutions of butyl alcohol and isobutyl alcohol exhibit this property.

In such solutions, separation at the surface must also take place in a definite temperature region and disappear on either heating or cooling. The region of surface separation may be displaced to a considerable extent with restrictivity coefficients of the components in the surface film differ considerably from the corresponding values within the volume of the solution. For this reason, surface separation must also occur in solution systems which remain completely homogeneous at all temperatures that have been encountered.

In the case of the system water-normal butyl alcohol which has been investigated, the n-butyl alcohol boiled at 117.4° (758 mm). Its density (d_{1}°) was 0.8007. The values obtained by measuring the surface tension of the components and their mixtures are shown in Table 1 see appended tables. The curve for % by volume of butyl alcohol could be traced to the temperature at which the solution separates into two layers. As the temperature is raised, this curve approaches that for pure butyl alcohol. The difference $\Delta G = G - G$ (and consequently also the surface activity $G = \Delta G$) increases as the temperature goes up (G) is the surface tension of pure water). It follows from this that here the adsorption must drop as the temperature. This contradicts the postulate that adsorption must drop as the temperature increases, because increased heat motion of molecules must lead to an equalization of concentrations between the surface film and the bulk of the solution (15). We shall attempt to explain this contradiction below.

For the temperature curves of a number of solutions, rapid changing of the coefficient y = 20 with the temperature is characteristic. At low temperatures, this coefficient is very high, greatly exceeding in magnitude the value of y for pure water, but within the span of a few tens of degrees drops practically to zero. The curves have been brought up to only 50° because above this temperature measurements of surface tension proved impossible because of fluctuations in the maximum pressure at which bubbles were formed.

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A. B. Taubman (16) regards the possibility of measuring of by establishing the maximum pressure at which formation of bubbles takes place as an index of the homogeneity of the surface film. He arrives at the conclusion that in the homologous series of alcohols the individual carbinols beginning with octyl alcohol form micelles in the surface film, and that for this reason accurate measurements of the surface tension become impossible.

One may assume that what exerts an influence here is not merely formation of micelles taking place as a consequence of separation, but also the tendency of these micelles towards coacervation. Formation of a surface emulsoid which has a high degree of dispersion and at the same time possesses sufficient stability may make the measurement of a rather difficult. Still, if certain conditions are observed, the resulting values are entirely reproducible. This must have been the precise state of the surface film in a number of organic systems showing surface separation that have been studied by us (11, 21).

Taubman's conclusions evidently refer to conditions at room temperature. Above 50°, as has been mentioned above, measurements of proved impossible even in the case of butyl alcohol. Consequently, from the standpoint mentioned above, a homogeneous surface film becomes non-homogeneous when the temperature is raised. In other words, surface separation takes place (this separation exhibits a lower critical point). At still lower temperatures, the degree of non-homogeneity of the surface film is reduced, because rormation of hydrates takes place. The reason for the increase in the value of a which takes place then is either that the hydrate has a higher surface tension than the pure alcohol or that because of stronger interaction between the components in the surface film, the molecules of alcohol, which are in excess there, draw into the surface film an additional

It is of interest to clarify the dependence of the temperature coefficient on the concentration of the solutions. We calculated the values of χ at two temperatures (10° and 40°) as tangents with the opposite sign of the slope at the points in question on the temperature-surface tension curve. The values of χ obtained in this manner are plotted in Figure 1 (see appended figures). The curves χ (c) pass through a maximum. On the 40° curve, the maximum is preceded by a minimum.

We assume that the raxima on the (c) isotherms are due to interaction between the components in the surface film (10). Reduction of the height of the maximum along with increases in the temperature indicates weakening of this interaction. Entirely similar curves for aqueous solutions of r-caproic acid were obtained by us on the basis of P. A. Rebinder's data. (17). These curves are shown in Figure 2. They indicate that similar relationships obtain in the case of n-caproic acid.

The system water-isobutyl alcohol was also investigated and found analogous to the system discussed above. The measurements were carried out with alcohol with a boiling point of $108^{\rm O}$ (at normal pressure) and the density ${\rm d_1^{\rm O}}_{\rm c}$ = 0.8021. The data are shown in Table 2.

At temperatures higher than $+45^{\circ}$, exact determinations became impossible for the reason stated above. The curves corresponding to 9.5 volume % and 10 volume % of isobutyl alcohol could be orought to temperatures at which volume separation takes place. As the temperature is raised, these curves approach the curve for pure isobutyl alcohol.

Just as in the case of the system discussed earlier, curves for solutions of isobutyl alcohol exhibit a very high value of the temperature coefficient. Thus, the 3% solution in the range minus 50 to plus 50 has a temperature coefficient of 0.31, while the 10% solution has a temperature coefficient of 0.35. The corresponding value for pure isobutyl alcohol is only 0.09 and that for water 0.14

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(ergs per square centimeter per degree). However, as the temperature rises, the coefficient χ , which remains constant in the beginning, drops rapidly, so that in the case of solutions with low concentrations (3%), its value approaches zero. The general shape of the curves indicates that at still higher temperatures χ may assume a negative value. We have shown earlier that abnormally low values of χ are characteristic for surface separation with an upper critical point. For that reason, one may assume that the final result of increasing

We regarded it as essential to carry out measurements on lower homologues as well. The aqueous solutions of these homologues do no show any separation.

the temperature must be transformation of the state of surface separation into a state corresponding to the formation of a completely homogeneous surface layer.

In the investigation of the system water-normal propyl alcohol the n-propyl alcohol used had a boiling range of $97.1-97.6^{\circ}$ at 756 mm and the density $(a_6^{\circ}0)$ of 0.8043. The surface tension in the temperature range minus 10° to plus 50° was measured. Measurements at still higher temperatures could not be made. The results of the measurements are shown in Table 3.

The temperature curves of solutions containing 0.5-20 volume % of n-propyl alcohol are curvilinear. Their convex side is turned toward the temperature axis. With the exception of the 40% curve, which has a small curvature at low temperatures, all remaining curves are practically rectilinear. The course which these curves assume is the same as in the case of the system that contains ethyl alcohol. This system is described below. Using the temperature coefficients determined by us graphically (for instance those at 250), we find on passing from pure vater over a number of solutions to pure n-propyl alcohol that the coefficients first pass through a minimum (for an 0.5% solution this lies at 0.135), then rise up to a maximum value (0.192 for a 10% solution), then drop again and, on passing through a low minimum (0.06), finally rise and approach a value corresponding to that for pure n-propyl alcohol (0.064 ergs per square centimeter per degree). We must note that when the temperature drops, the maximum value of rises and approaches in magnitude the corresponding values in preceding systems.

In the investigation of the system water - ethyl alcohol, the ethyl alcohol had a b. pt. of 78.0° at 757 mm and the density (d_4^{20}) of 0.7898. Measurement of surface tensions in this system was carried out in the temperature range minus 10° to plus 60° . The results of the measurements in question are given in Table 4.

The values found for \leq at 25° were in good agreement with published data (2, 14, 18).

Examination of the curves obtained in this case shows that some of them are rectilinear, while others are smooth curves. One may regard as rectilinear the curves for solutions containing less than 3% of alcohol and those for all solutions containing more than 40% of alcohol. Solutions of intermediate concentrations give curvilinear graphs. It is characteristic for the latter solutions that the coefficient acquires in the region of low temperatures a value which is higher than that of the components. In this respect they resemble the systems discussed previously.

We consider it expedient to regard $\frac{\delta}{\delta}$ as a function of the composition of the surface layer. Many investigators, on the basis of a study of surface tension isotherms, made various assumptions in regard to the structure and composition of surface films in the system water - ethyl alcohol (3-8). However, this problem cannot be regarded as completely solved. The theory of A. A. Zhukhovitskiy (8), according to which the surface film is regarded as an ideal solution, permits calculation of the composition of the surface film. The values obtained as a result of this calculation may be regarded as a good approximation: they differ essentially from the data computed by Butler, Wightman (4) and Guggenheim, Adam (5).

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In Figure 3, the temperature coefficients of the system at 25°, which we calculated by using the graphic method, are plotted against values giving the composition of the surface film which have been calculated on the basis of A. A. Zhukhovitskiy's data. The curve of the temperature coefficient drops starting with the 10% point for each of the two components and, on passing through a minimum, rises to the maximum value of χ . The maximum deviation of χ from the value corresponding to a purely additive effect occurs at about the 50% point. This means that the maximum of surface entropy corresponds to an equimolar ration of components in the surface film. The curve also indicates that χ composition of the hydrate.

We may note that the idea in regard to the hydration of molecules which are oriented in the surface film of a solution has been advanced earlier (19, 20).

In the investigation of the system water - methyl alcohol, methyl alcohol, distilled at 64.50 (757 mm) after drying, was used. Its density (d_k^{20}) was in Table 5.

At first glance, these results seem to be different from those obtained in the case of ethyl alcohol: the temperature curves are practically rectilinear. Using the method of the least squares, we determined the equations representing these curves (cf the last column of Table 5). All deviations of experimentally determined points from the points disposed on the straight lines are accidental and do not exceed the limits of experimental error.

The angle coefficient of the temperature curves (with a reverse sign), i.e., the temperature coefficient of passes through a minimum. If the values of are plotted along the axis of ordinates, and the volume or molar fractions of alcohol contained in the solution along the axis of abscissae, there is only a very small amount of dispersion at low concentrations. If the molar fractions of alcohol in the surface film (as found, for instance, on the basis of A. A. Zhukhovitskiy's data) are plotted along the axis of abscissae, we obtain the curve shown in Figure 4. This curve indicates the essential similarity between the system under consideration and the preceding system. The difference consists in the fact that in this particular case positive deviations of from additivity are not great enough to give rise to a maximum on the curve. The general character of the curve is essentially the same, however. It is noteworthy that in this system as well the maximum deviation from additivity corresponds to a composition of the surface layer which lies close to an equimolar proportion of the components. Thus, in this system the shape of the temperature coefficient curve also indicates the formation of hydrates in the surface film.

We acknowledge the active participation of Academician P. A. Rebinder and Prof A. B. Taubman in the discussion of the work published in this instance.

Conclusions

- Determination of the surface tensions of aqueous solutions of methyl, ethyl, n-propyl, n-butyl, and isobutyl alcohels was carried out at various temperatures.
- Consideration of the temperature coefficients of the surface tension in the systems studied permits the conclusion that hydrates of alcohols are formed in the surface film of the solutions in question.

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Table 1.	Surface	Tensions	of	the	System	Water	-	n-Butyl Alcohol	
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% C4F	E ² OH						J		PÁCAT WICO	101			
<u>.</u>	mol_	_00	_ <u>5</u> °	<u>10°</u>	_15°	200	250	300	_35°_	<u> 100</u>	450	50°	
0	0	75 • 59	74.88	74.19	73.50	72.75	72.01	71.18	70.41	69.90	68.78	67.93	
0.1	0.02	72.84	72.07	7.138	70.61	69.84	68.91	68.14	67.30	66.61			
0.4	0.08	66,60	65.46	64.53	63.60	62.68	61.91	61.11	60.52		65.91	65.2 9	
1.0	0.20	58.05	56.86	55.60	54.72	53.81	52.80		•	59.85	59.29	58.92	
1.5	0.30	53.73	52.4 9				-	52.03	51.41	50.85	50.40	50.25	
	0.40	• • •	•	51.02	50.02	49.18	48.17	47.17	46.60	46.02	45.47	45.24	
2.0	• •	50.03	48.78	47.47	46.37	45.54	44.57	43.82	43.19	42.57	42.27	41.90	ım
3.0	0.61	46.37	44.90	43.43	42.02	40.57	39.74	38.91	37.89	37.43	36.97		8-1-C-R-18-1
5.0	1.02	39.65	37.98	36.51	34.85	33.48	32.63	31.70	30.98			36.69	녆
6.0	1.23	36.97	35.40	33.84	32.36	31.16	_			30.43	29.93	29.59	牍
8.0	1.67	32.73	30.98				29.87	29.04	28.49	27.84	27.20	26.83	1-3
9.0	1.89			29.59	28.40	27.40	26.37	25.55	24.80	24.25	23.88	23.69	
	-	30.98	29.59	28.21	27.20	25.91	25.07	 .					
100	100	26.29	25.82	25.36	24.90	24.52	23.98	23.51	23.13	22.74	22.36	22.05	

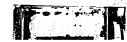
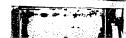


Table 2.	Surface Tensions	of the	System Water	- Isobutvi A	Icohol
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	% C1	II-OH								TCOHOL				
	o Ā	<u>mol</u>	<u>-5°</u>	0° 75.70	+50	100	150	200	250	300	<u>350</u>	400	heo	
	3	0.61	46.36	44.71	74.96 43.26	74.27 42.08	73.51	72.75	71.98	71 21	70.37	69.52	450 68.76	
	5	1.02	40.91	39.66	38.21	36.97	40.94 35.51	40.08 34.41	39.32	38.56	38.01	37.52	37.42	
	6 8	1.23	39.18	37.87	36.14	34.83	33.31	32.20	33.58 32.37	32.82 30.54	31.99	31.51	31.30	
lo in	9	1.67 1.89	35.03 33.44	33.86 31.92	32.13	30.82	29.51	28.54	27.43	26.60	29.85 25.91	29.30 25.29	28.81 25.01	
E-E-U-E-E	9.5	2.01	32.96	31.02	30.06 29.30	29.02 28.26	27.78	27.02	25.98	25.08	24.46	23.91	23.56	ΙĖ
i+3 M	10	2.12	32.41	30.68	28,95	27.64	26.88 26.33	25.57 25.15	24.53	23.63	22.94			8-8-0-8-8-1
	100	100	24.74	24.39	23.84	23.42	23.08	22.73	23.98 22.46	22.04	 21.63	 21.35	 21 -01	igi igi



J. Duriace Tensions of the System Water - n-Propyl Alcohol

	%	с ₃ н ₇ он							DARGE ME	ater - n-	Propyl Ale	cohol				
	<u>. v</u>		<u>-100</u>	<u>-5°</u>	_ 00_	<u>+ 50</u>	100	15°	200	25°	30°	250	Leo			
	0	0			75.70	74.96	74.27	73.51	72.75			350	400	450	500	
	0.5	0.1			70.58	69.38				71.98	71.21	70.37	69.52	68.76	67.92	
	1.0	0.2					67.77	67.21	67.21	66.30	65.87	64.96	64.32	63.76	63,20	
					65.19	64.13	63.01	62.16	61.32	59.68	59.91	59.00	58.50		-	
	2	0.5		62.66	61.11	59.84	58.92	57.80	57.09	56.18				57.94	57.52	
	5	1.2		53.43	51.46	50.48	48.93	47.80	-		55.48	54.84	54.21	53.72	53.36	
ro.	10	2.6	46.18	44.42	42.66	41.11			46.60	45.90	##*8#	44.14	43.65	43.02	42.80	
ka .	20	5.7	2), 20				40.13	38.86	37.88	36,68	35.90	35.13	34.50	34.14	22 11	
0				32.81		30.48	29.71	28.86	28.30	27.67	27.24	27.00	-	•	33.44	In Ito
a ;	40	13.8	27.74	27.32	26.96	26.54	26.19	26.05	25.77	25.34		-	26.68	26.40	25.91	10 10
3	60	26.6	27.24	26.96	26.54	26.19	25.77				25.13	24.71	24.57	24.22	23.80	늄
	30	49.1		26,75				25.48	25.20	24.78	24.36	24.11	23.90	23.55	23.20	117
	100	100				25.94	25.62	25.18	24.87	24.43	24.04	23.65	23.37			
	100	100	20.26	25.80	25.48	24.92	24.57	24.15	23.79	23.23	22.92	22.16	-2-21	22,92	22.53	

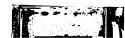
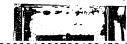


Table 4. Surface Tensions of the System Water - Ethyl Alcohol

%	C2H50	H										•				
	mc	1 -10	-50	00	+5°	100	15°	200	250	300	35°	400	45 ⁰	50°	550	<u>60°</u>
0	_			12.1	0 14.9	0 74.2	7 73.5	1 72.79	71.9	8 71.2	1 70.3	7 69.5	2 68 7	6 67 0	2 62 4	
0	.5 0.	18		72.8	1 72.16	71.3	L 70.58	69.86	69.08	68.2	2 67.5	7 66 8	= 66 1	0 01.9	2 67.0	5 64.16
0.	75 0.2	<u>2</u> 6		72.09	71.31	70.65	70.13	69.27	68,55	67.76	67.15	, 66 1.	. (= 0	2 65.5	3 64.7	5 64.15 5 63.50
1.	0 0.3	S5	72.29	71.70	70.78	70.00	69.34	68.81	67.00	67.10		00.4	05.80	64.88	64.16	62.98
3.	0 1.2		66,65	65.87	65.08	64.18	62 27	60.02	(2.50	01.11	00.33	65.73	64.94	64.09	63.57	62.98
5.	0 1.8		63.22	62.hh	61.52	60.20	50.51	02.50	61.73	61.60	60.35	59.76	59.10	58.38	57.86	57-33
10	3.5		56.20	Eli Oli	52.02	00.39	59.47	58.55	57.70	56.96	56.26	55.56	55 • 57	54.33	53.71	57.33 53.24
20			وعودار	J4.94	23.01	52.68	51.75	50.83	50.06	49.21	48.57	47.79	47.29	1:6.59	46.16	53.24 45.56
	11.3		₩.51	47.31	44.32	43.40	42.34	41.35	40.71	39.93	39.01	38, 51	27 88	25 50		
		39.29	30.30	31+72	30.90	30.32	35.47	35.12	34.55	33.91	33,42	33.06	22 57	20.34		
	17.2	33.10	33.37	33.00	32.64	32.20	31.86	31.36	31.01	30.68	30,30	29.81	20 74	00 10	-0 0-	
60	31.6	30.00	-9.0 1	29.30	29.03	25.67	28.25	27.97	27.68	27,19	26.97	26 1/8	26 27	05 01		
80	56.1	27.90	27.54	27.04	26.69	26.41	25.98	25.70	25,13	24.85	oh ha	2b 07	00.21	27.04	25.70	25.28
90	74.5	26.50	26.11 s	25.67	25.26	24.86	24.46	23 88	->	02.00	24.43	24.07	23.72	23.22	22.87	21.37
100	100	24.88	24.49	23.94	23.6h	22 17	20.70	~	-3.77	~3.22	22.76	22.44	22.04	21.58	21.12	20.80
			24.49	5-5.	-5.04	-3.11	£2.10	22.32	21.86	21.54	21.00	20.69	20.23	19.84	19.38	18.99

50X1-HUM

S-E-C-R-E-T



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Table 5. Surface Tensions of the System Water - Methyl Alcohol

		%	изон										-				
		, <u>v</u>	mol	<u>-10°</u>	-50	00	<u>+5°</u>	10°	15°	20°	25°	30°	35 ⁰	400	450	500	Equations of the Curves
		0	0			75.70	74.96	74.27	73.51	72.75	71.98	71.21	70.37				<u>d</u> =75.80+0.158 t
		2	0.9		72.16	71.37.	70.78	70.16	69.40	68.78	68.02	67.41	66.64		-		<u>6</u> =71.47-0.138 t
		5	2.3		67.41	66.87	66.03	65.72	64.88	64.34	63,50	62.89	62.20	61.59	60.82	60.21	d' 66 05 0 200 .
			, .		61.82	61.13	60.51	59.67	59.14	58.60	57.76	57.22	56.61	55.84	55.15	54.60	6=61.14-0.131 t
				54.15	52.61	52.80	52.32	51.65	51.17	50,63	50.02	49.48	49.01				₫=52.92-0.115 t
ក្រ លៃ		30	16.1	46.71		45.81								•			<u>Ø</u> =32.92-0.115 t <u>Ø</u> =45.81-0.092 t
-C-R-E	Ë	50	31.0	38.67	38.24	37-75		36.97									
i	•	70	51.2	31.60	31.11	30.75	30.40	29.84					27,71				<u>d</u> =37.79-0.084 t
114		100	100	25.40	25.03	24.44	23.91	23.50	23.05	22.50							₫=30.73-0.086 t 150
									_3,		22.09	<1.71	21.21	20.67	20.29	19.90	ď=24.44-0.093 t 113

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 $\underline{S} - \underline{E} - \underline{C} - \underline{R} - \underline{E} - \underline{T}$

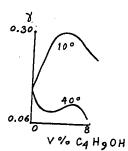
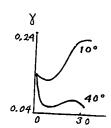


Figure 1. Temperature coefficients of the surface tension of aqueous solutions of n-butyl alcohol.



Millimol/liter of C₂ H₁₁ Cool H

Figure 2. Temperature coefficients
of the surface tension
of acqueous solutions of
caproic acid (according to
P. A. Rebinder's data).



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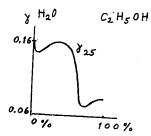


Figure 3. Temperature coefficient of surface tension in the system water-ethyl alcohol. Molar fractions of alcohol in the surface film are plotted along the axis of abscissae.

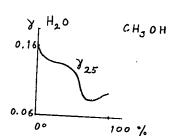


Figure 4. Temperature coefficient of surface tension in the system water-methyl alcohol. Molar fractions of alcohol in the surface film are pletted along the axis of abscissae.

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- 12 -

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